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Technical Report

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STUDIES OF DEFORMATION IN NIOBIUM BY X-RAY TOPOGRAPHIC METHODS

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Summary

We have studied plastic deformation in niobium crystals by a variety
of x-ray topographic techniques including: Lang topography, double
crystal topography and synchrotron white beam topography. The
limitations of these direct methods, which rely on the resolution of
individual dislocation segments, are illustrated by our results. An
indirect topographic method, contour mapping, is described for use with
specimens which cannot be profitably studied with the direct
techniques. The positions of equi-inclination contours (analogues to TEM
bend contours) are recorded as a function of specimen rotation, allowing
measurement of the components of the strain tensor as a function of
position on the specimen. Use of monochromatic radiation and of
synchrotron white radiation are described and results obtained with
contour mapping are summarized.

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Introduction

The special vantage point offered by x-ray diffraction topography is best illustrated by comparison with a more commonplace technique, transmission electron microscopy. Both techniques rely on image formation via diffraction contrast; because of the nature of the diffracting entities, the most rewarding applications for each are different. One obvious difference is the ease with which electron or x-ray beams may be focused: effective lenses exist for the former but are not available for the latter.

The advantages of x-ray diffraction topography include very high sensitivity to strain and much greater penetrating power compared to TEM. Topographic examination of a specimen does not require destructive sample preparation, and topography allows a single specimen to be examined many times during the course of an experiment. Large volumes of material may be studied although relatively long exposure times (2-48 hours) are required for high resolution topographs recorded with laboratory sources of x-radiation. Mainly because of its high sensitivity to strain, the spatial resolution of topography is quite low. As a result, dislocation images on x-ray topographs have minimum widths of about one micrometer, and the lack of an effective lens for x-rays does not prevent the use of topography as a characterization technique. Images of individual dislocations can only be obtained from specimens (crystals or poly-crystals with grain diameters greater than about 100 μm) with dislocation densities $\rho < 10^6$ cm^{-2}. X-ray diffraction topography is, therefore, used to the best advantage in studying long range strain fields in relatively dislocation free crystals, and it is not surprising that the primary use of x-ray topography has been to characterize crystal growth defects.

The advent of synchrotron radiation sources and of rapid imaging systems for laboratory sources has shifted emphasis to x-ray topographic observation of dynamic experiments. In the context of this volume, in situ studies of plastic deformation such as those of Miltat and Bowen (1) and Michot, George and Champier (2) are of particular interest. The arrangement and propagation of dislocations under stress were observed by these authors, but only the earliest stages of plastic deformation could be examined if individual dislocations and slip bands were to be resolved. While the direct or conventional x-ray topographic techniques (those which depend on dislocation imaging) have limited applicability, the indirect topographic methods (those which map deformation fields without forming dislocation images) allow one to study quite heavily deformed material.

One indirect method is the point-by-point mapping of rocking curve widths (3). This technique is particularly attractive because poly-crystals as well as single crystals may be studied. Rather large areas of the specimen (typically 200 μm x 60 μm) must be irradiated (4); and a significant amount of strain averaging may occur. Also, it appears that the process of extracting the individual components of strain is rather involved.

Another indirect topographic method uses equi-inclination contours, which are the x-ray topographic analogues to bend contours in electron microscopy. The patterns formed by rotating the specimen and recording many contours allow one to see directly the distribution of strain (5); and the spatial separation of contours produced by known specimen rotations have been used to measure the strain associated with uniform

bending of crystals (6). If one uses sets of equi-inclination contours, obtained with different diffraction vectors and rotation axes, all of the individual components of strain can be determined as a function of position (7,8). Analytic methods for both monochromatic and synchrotron white radiation sources have been developed, and the feasibility of this approach has been demonstrated by measuring some of the components of the strain field surrounding a precipitate of β-NbH.

In this paper we present x-ray topographs which are representative of our studies of plastic deformation in the niobium-hydrogen system. Results obtained with imaging techniques and with equi-inclination contour mapping are used to illustrate the advantages or shortcomings of each approach. The principal advantages of x-ray topography should be remembered throughout: high strain resolution coupled with non-destructive observation of large volumes of the specimen. It should be added that all results reported below were obtained in transmission and that Mo Kα radiation was used for all topographs recorded with laboratory x-ray sources.

Imaging Methods

A number of imaging techniques were used to study plastic deformation in niobium crystals. Lang topography was used to examine as-grown and slightly deformed crystals. Double crystal topography, using characteristic radiation, was also employed to observe as-grown crystals and to follow plastic deformation at notches during tensile tests. Synchrotron white beam topography was the third technique employed, and it appeared to be the most satisfactory for imaging dislocations, slip bands, etc., which result at low strain levels.

Lang Topography

The Lang technique is probably the most widely used x-ray topographic method. An image of the specimen is obtained by recording the diffracted beam of x-rays on a piece of film. The incident beam is collimated to a narrow ribbon on the order of 150 μm wide by 15 mm high so that reasonable spatial resolution may be obtained. Large volumes of the crystal are observed by scanning the specimen and film in tandem across the stationary beam of x-rays. Special photographic emulsions with small grain size and large thicknesses (20 to 50 μm) are used to record the image of the diffracted beam, and optical microscopy provides magnified views of the topograph.

Figure 1a is a topograph of a 75 μm thick niobium crystal recorded with h = [1̄10]. The slightly bent crystal shows orientation contrast from different subgrains (labeled A and B) and from part of the crystal at which the bending has produced a misorientation greater than the range of angles which the crystal can diffract. Diffraction contrast from low angle boundaries and from scratches scribed on the surface (features C and D, respectively) appears as darkening on the topograph.

A topograph of a notched niobium crystal of better quality (lower initial dislocation density and curvature) is shown in Figure 1b after a small amount of tensile loading. The 125 μm thick crystal was deformed in a small tensile stage, and the specimen was carefully removed from the grips of the stage prior to recording the h = [112] topograph pictured here. Orientation contrast from different subgrains (features such as S3 and S4) is prominent. Most of the specimen's volume is oriented for

diffraction except that part near S4 and that in the interior of the plastic zone near the notch, labeled N. An increase in diffracted intensity is observed from the plastic zone allowing one to measure its size.

It is possible to image the entire volume of a bent or deformed crystal by rotating it during the exposure of the film. Our experience has been that the required rotations lead to intolerable broadening of dislocation images unless the plane of the specimen intersects the rotation axis.

Double Crystal Topography

Double crystal topography is more sensitive to smaller strains than is the Lang technique, and it would not seem to be suitable for imaging dislocations resulting from deformation at a notch. With this technique, a reference crystal or monochromator diffracts the x-radiation from the source and provides a parallel beam with which to study the specimen crystal. An asymmetrically cut silicon (111) reference crystal was used in our experiments to form a spatially wide, yet parallel, beam. Less volume of the crystal will diffract at any particular orientation than with Lang projection topography: many equi-inclination contours, however, may be recorded on a single piece of film, by successive rotations of the specimen. An image of the entire crystal is formed, and little image broadening will occur since the images contained in each contour will be visible only at one orientation. A small tensile stage, patterned after that of Bowen and Miltat (10), was used for in situ straining of the crystals.

Examples of double crystal topographs of notched specimens after small amounts of strain are shown in Figure 2. Both topographs are of the same specimen as was shown in Figure 1b. The topograph in Figure 2a was taken before the crystal was mounted in the tensile stage. The diffraction vector $\underline{h}$ = [121], the rotation axis [1$\bar{1}$1] and rotation increments of 105 arc sec. were used to record the contours which form this topograph. Subgrains S1 and S3 are not oriented for diffraction, but subgrain S2 is, and an image of S2 is superimposed on the image of the largest subgrain. Damage, introduced by spark machining of the

Figure 1 - Lang projection topographs of niobium single crystals. The diffraction vector is indicated by $\underline{H}$. Both topographs show orientation and extinction contrast. The first crystal is as-grown, and the second was notched and was loaded in tension along the horizontal axis.

notch, is visible around it despite chemical polishing which removed at least 25 μm of niobium. The spark machining was performed so as to minimize this damage. It is apparent that extreme caution must be exercised when studying specimens which have been fabricated by spark machining, even if more than 100 μm of material has been chemically removed. Figure 2b,c are topographs of the crystal after different amounts of strain. They were recorded with $\underline{h}$ = $[002]$ and with rotations of 105 arc sec. about axis $[1\bar{1}0]$. Discussion of the features in this topograph will be postponed until the section on equi-inclination contour mapping.

White Beam Topography

Synchrotron radiation is ideal for use with x-ray topography (11,12): the resulting parallel beam of white radiation has an extremely high intensity in the range of wavelengths normally used for topography There are considerable advantages to using white radiation since slightly deformed crystals, such as those in Figures 1 and 2, can diffract over their entire volumes simultaneously. Each misoriented region will select a slightly different wavelength for diffraction, and a number of topographs may be recorded with a single exposure, as shown in Figure 3. Figures 3-5 were obtained during in situ deformation studies conducted at the Daresbury Synchrotron Radiation Source (13). These experiments were observed in real time using an x-ray television camera (14), but its relatively low resolution made it desirable to record the topographs on Ilford L4 nuclear emulsions.

Figure 4a-c are white beam topographs of a niobium crystal which had been charged with 1.8 at.% hydrogen. The dislocation density of the crystal shown here and that shown in Figure 2 were similar. Use of white radiation allowed the entire region of interest to be observed at one time. Figure 4a was recorded with $\underline{h}$ = $[1\bar{1}2]$ and shows the plastic zone beginning to extend from the notch (labeled N). As grown features included individual dislocations and remnants of dislocation tangles which had not been removed by annealing. Considerable asterism is visible along the edges of the notch; it is a result of the severe rotations introduced by spark machining the notch. Figure 4b is an
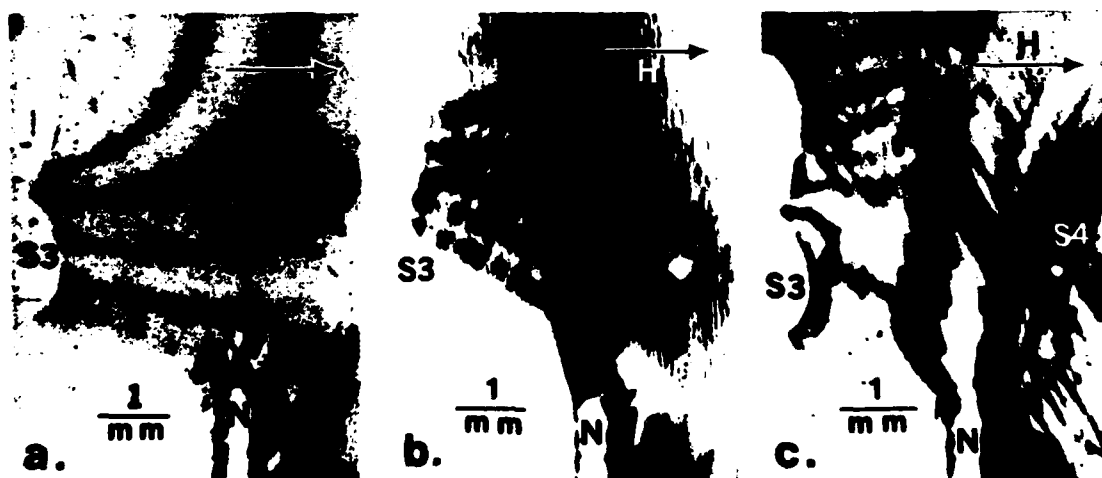

Figure 2 - Double crystal topographs of a notched niobium crystal after different amounts of deformation. a. $\underline{h}$ = $[121]$. b. $\underline{h}$ = $[002]$ and c. $\underline{h}$ = $[002]$.

enlargement of Figure 4a. The plastic zone and slip bands, which were extended on one side of the plastic zone, are shown although the asterism from the notch obscures some details of the slip bands. Individual dislocations, which apparently accommodate the slight curvature of the specimen, can be seen ahead of the plastic zone. After the plastic zone moved across the specimen (Figure 4c, at a nominal stress of 13.9 MPa and with the same diffraction vector as Figure 4a, b), no usable images remain. In most experiments, however, this strain would be considered to be rather low.

The topographs of Figure 5 are of a crystal which had been loaded in compression prior to the tensile deformation at 150 K. Severe asterism (Figure 5a) is visible in the region deformed in compression, and it is interesting to note that this region is quite narrow. With white beam topography no information can be gathered from this region, although the lateral spread of the plastic zone from this region during tensile

Figure 3 — White beam topographs of a niobium crystal. The images are individual topographs with lighter regions correspond greater diffracted intensifies.
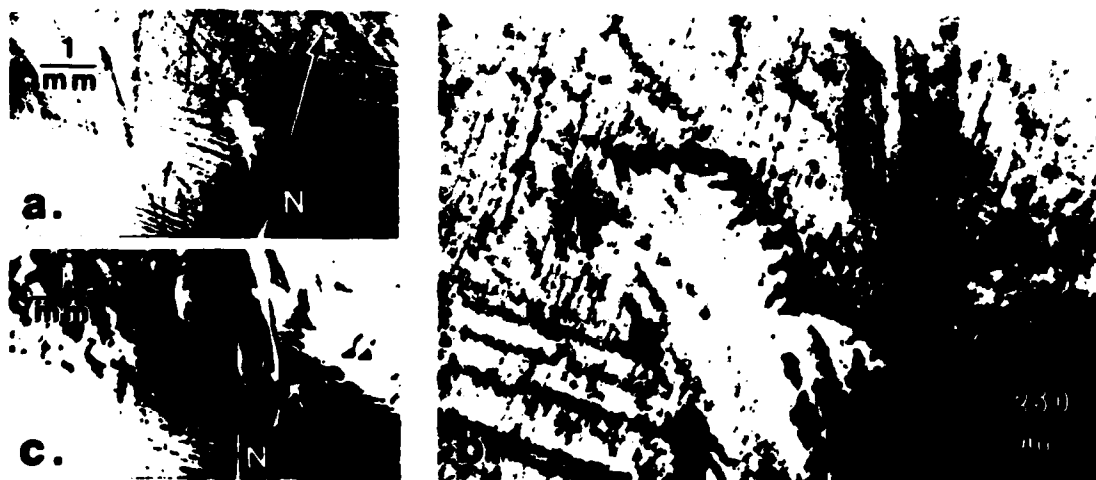

Figure 4 - Topographs of a notched Nb - 1.8 at. % H crystal recorded with $h = [1\bar{1}2]$. a. Early stages of plastic zone extension. b. Severe deformation later in the tensile test. c. Enlargement of the plastic zone shown in a.

loading can be followed. This type of imaging is helpful in studying the transfer of slip across subgrain boundaries (Figure 5b, c). In Figure 5b the image of the subgrain has rotated, reflecting the change in orientation due to bending near the notch, and in Figure 5c slip bands are interacting in the interior of this subgrain. One must be wary, therefore, when interpreting white beam topographs: a range of diffraction angles are possible and significant artificial shifts of different parts of the image may occur.

Contour Mapping

While a qualitative estimate of the deformation is useful, as illustrated by the double crystal topographs, the power of contour mapping comes from its use in measuring strain components ϵ_{ij} as a function of position. The formalism is given below by which ϵ_{ij} is related to the contour spacings and the corresponding specimen rotations. Methods for separating contributions from the shear and dilational strains are presented for the cases of monochromatic radiation and of white radiation. The experimental results which have been obtained with this technique are also discussed, and improvements for future measurements are suggested.

Consider a strain center which deforms a thin, ribbon-like single crystal and which is centered on a rectilinear coordinate system $\underline{x}_1$ with $\underline{x}_2$ normal to the largest face of the crystal. Diffraction from planes normal to axis $\underline{x}_1$, denoted by P_1, is considered for the case of a parallel, spatially-broad beam of monochromatic x-rays. As a result of the non-uniform deformation, only a small fraction of the crystal diffracts at any single orientation of the x-ray beam relative to the crystal. If the initial region oriented for diffraction is denoted by ℓ, the specimen rotation required to correctly orient a region ($\ell+1$) is defined as

$$\omega_1^k(\ell+1, \ell) = \Theta(\ell+1) - \Theta(\ell), \qquad (1)$$
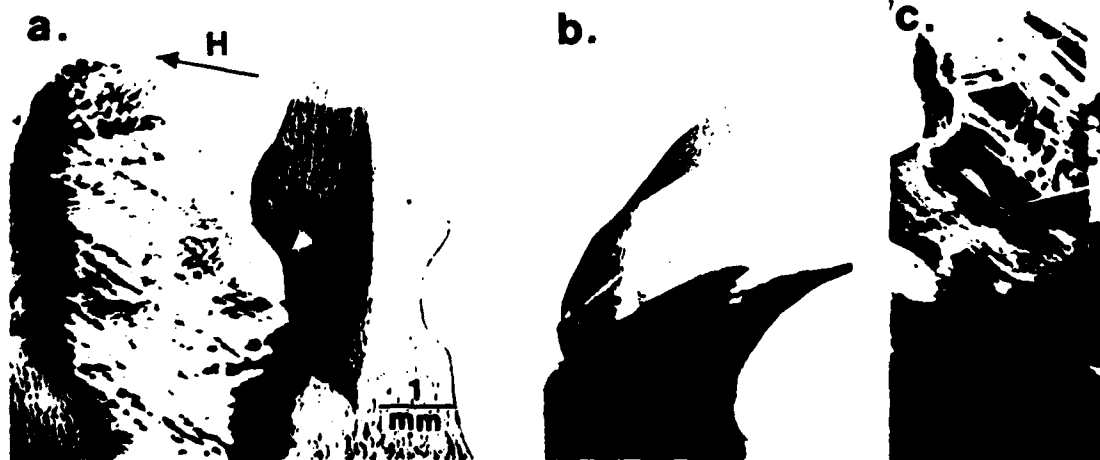
Figure 5 - Three stages of deformation at 150 K in a notched niobium crystal. The crystal was deformed in compression before the tensile test. Slip transfer across subgrain boundaries is illustrated.

where $\Theta(\ell)$ and $\Theta(\ell+1)$ are the angles of incidence of the x-ray beam for diffraction at positions ℓ and $\ell+1$, respectively; the superscript k denotes the rotation axis $\underline{x}_k$ and the subscript i the diffraction plane P_i. Noting that only deformation with components normal to P_i can be observed, we can relate the rotation to the derivatives of displacement with respect to distance by

$$\omega_i^k (\ell+1,\ell) = \frac{\partial u_i(\ell+1,\ell)}{\partial x_m} - \frac{\partial u_i(\ell+1,\ell)}{\partial x_i} \tan \Theta_0 , \qquad (2)$$

where Θ_o is the Bragg angle for diffraction of the monochromatic radiation by planes P_i. The first term is part of the shear strain ε_{im} and represents the change in the amount of tilting of planes P_i about axis $\underline{x}_k$, and the second term is the contribution of dilational strain ε_{ii}. In the above expression $\underline{x}_i \neq \underline{x}_k \neq \underline{x}_m \neq \underline{x}_i$, and the contribution from $\partial u_i / \partial x_k$ is negligible (7,8).

The limited volume of material diffracting at each angular setting produces narrow bands of darkening on topographs of deformed crystals, and the position of an equi-inclination contour may be mapped as a function of crystal rotation using multiple exposure of a single piece film or a set of singly exposed emulsions. Characteristic radiation or monochromatized synchrotron radiation may be used to produce these contours. Similar contours (termed absorption edge contours) may be produced with synchrotron white radiation by orienting the deformed crystal so that the range of wavelengths diffracted encompasses the absorption edge of an element in the specimen (8). The absorption edge contour corresponds to diffraction of radiation with the same energy as the absorption edge; it is quite prominent in x-ray topographs because of differing absorption coefficients on either side of the edge lead to a marked difference in darkening of the emulsion.

The individual terms $\partial u_i / \partial x_i$ and $\partial u_i / \partial x_k$ cannot be determined, however, from a single contour map. The additional information is obtained by recording a number of contour maps using: first and higher order diffraction from planes P_i, diffraction from P_i with two or more wavelengths or diffraction from planes P_i with entrance and exit faces of the crystal exchanged (8). Once the appropriate pair of contour maps are obtained for P_i, $\partial u_i / \partial x_i$ and $\partial u_i / \partial x_k$ can be determined as a function of position (x_1 , x_3). All of the $\partial u_i / \partial x_j$ may be determined by choosing the appropriate diffraction planes and rotation axes. The variation of strain in the direction $\underline{x}_2$, normal to the largest face of the crystal, cannot be determined by this approach since the transmission measurements average over the thickness of the specimen.

One application where the contour mapping technique is particularly valuable is the evaluation of the strain field surrounding a large precipitate in a single crystal matrix: sensitive measurement of strain can be necessary over distances as great as several millimeters. The deformation accompanying the formation of β-NbH is one example of a precipitate which has been extensively studied with electron microscopy

(15, 16). Precipitates of β-NbH can grow to sizes as large as 1 mm, however, and we know of no studies of the distribution of strain around such hydride precipitates.

A precipitate of β-NbH was grown from a niobium crystal containing about 3 at.% hydrogen by cooling in a temperature gradient. Relatively large precipitates were obtained (750 μm x 150 μm x 50 μm were the maximum dimensions), and some had no neighbors within several milli-meters. Lang topographs were used to select a suitable precipitate, and a typical topograph, recorded with $\underline{h}$ = [110], is shown in Figure 6a. Considerable deformation is evident, and the feature marked B is the image of the precipitate selected for further study. An example of the contour maps recorded around this precipitate is shown in Figure 6b, a multiple exposure topograph with $\underline{h}$ = [002] and specimen rotations of 210 arc sec. Deflection of the contours can be seen as far as 2 mm away from the precipitate, indicating an extremely broad deformation field. Two of the terms $\partial u_3/\partial x_3$ and $\partial u_3/\partial x_2$ were determined as a function of position (direction $\underline{x_3}$ was parallel to the largest face of the precipitate) and had comparable magnitudes near the precipitate (17). The dilational term, however, decreased more rapidly than did the shear term. In these measurements, spatial resolution was estimated to be about 10-20 μm . Details will be discussed elsewhere.

A second example of contour mapping, shown in Figure 2, characterizes the distribution of strain around the plastic zone at a notch in a niobium crystal. Bending is concentrated near the plastic zone, and the elastic interaction between it and the neighboring subgrain leads to contours which are closely spaced between the two features. Plastic deformation has accumulated adjacent to the small subgrain labeled S4. Additional contour mapping must be completed, however, before this technique's use for plastic zone characterization may be realistically evaluated.

Figure 7 shows several topographs of absorption edge contours recorded with $\underline{h}$ = [002] and rotations of 90 arc sec. Several slip bands have extended from the notch (labeled N) in this slightly bent niobium crystal. There is no deflection of the contour when it passes through the slip bands, indicating that the level of deformation is quite low or
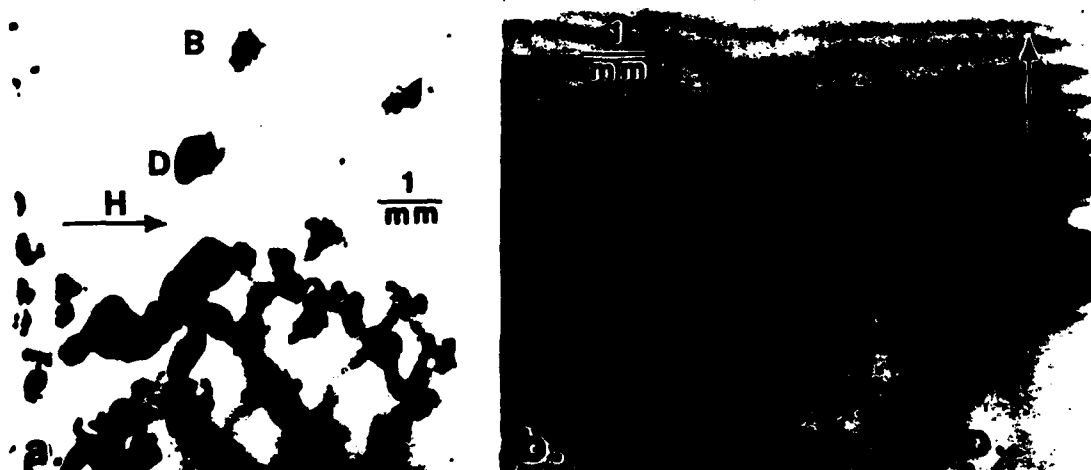

Figure 6 - Equi-inclination contours showing deformation around β-NbH precipitates. a. Lang projection topograph recorded with $\underline{h}$ = [110]. b. Multiple exposure double crystal topograph recorded with $\underline{h}$ = [002] and with specimen rotations of 210 arc sec.

that the observed contrast may be due primarily to the intersection of the slip bands with the surface.

It appears the absorption edge contour mapping is superior to equi-inclination contour mapping. In applications where the strain center has a complex morphology, like that of the hydride precipitate described above, one cannot precisely identify locations within the strain center when comparing equi-inclination contour maps with different diffraction vectors. Absorption contour mapping does not suffer from this difficulty because an image of the entire crystal is obtained each time a contour is recorded. Also, the relatively large strains within plastic zones formed at notches will broaden the equi-inclination contours appreciably, decreasing strain and spatial resolution. Absorption edge contours would presumably be superior in this application since the drastic change in absorption coefficient leads to a very sharp contour.

There is a third advantage to using absorption edge contours: one may simultaneously study chemical and crystallographic defects in the undeformed regions of the crystal (18). This approach relies on the fluctuations in the absorption coefficient as a function of wavelength on the high absorption side of the edge (termed EXAFS). A result is the formation of fringes of increased darkening in topographs of slightly bent crystals. If, for example, there is significant local segregation, deflection of the fringes would be expected. Unusual results obtained in subsequent experiments might then be attributed to the presence of such inhomogeneities.

Conclusions

The results of our studies of plastic deformation in niobium crystals show that x-ray topography can be very useful in this application. Imaging methods such as Lang, double crystal and synchrotron white beam topography were particularly useful in observing the earliest stages of plasticity in notched crystals. These techniques are, however, limited to specimens in which the dislocation density is less than about 10^6 cm^{-2}. More heavily deformed crystals, exemplified by that containing the β-NbH precipitates, can be studied by indirect x-ray topographic techniques, such as equi-inclination mapping. Our studies of plastically deformed specimens using equi-inclination contour
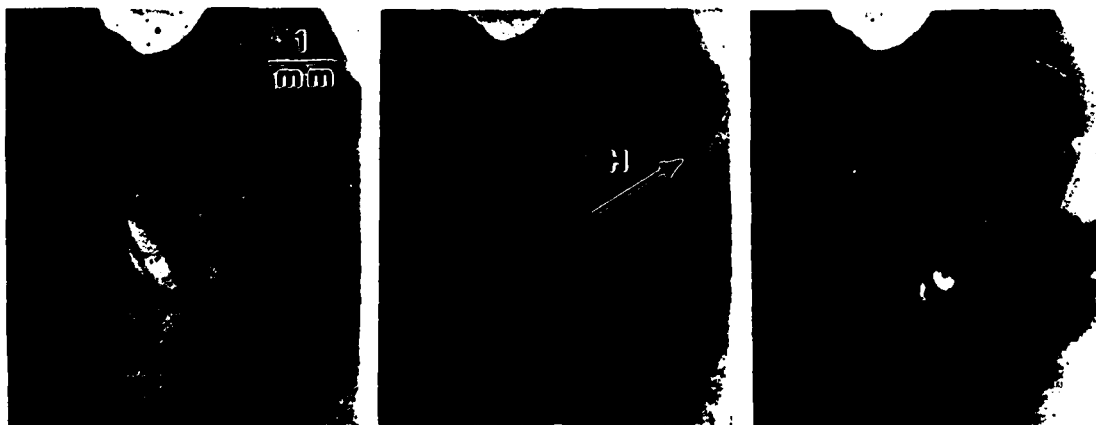

Figure 7 - Absorption Edge Contours of a notched niobium crystal after a small amount of tensile strain. Rotations of 90 arc sec. and $\underline{h}$ = [002] were used.

mapping to measure components of the strain tensor ϵ_{ij} indicate that it is a promising technique, combining reasonable spatial resolution (10 - 20 μm) with sensitivity to very small strains.

Acknowledgements

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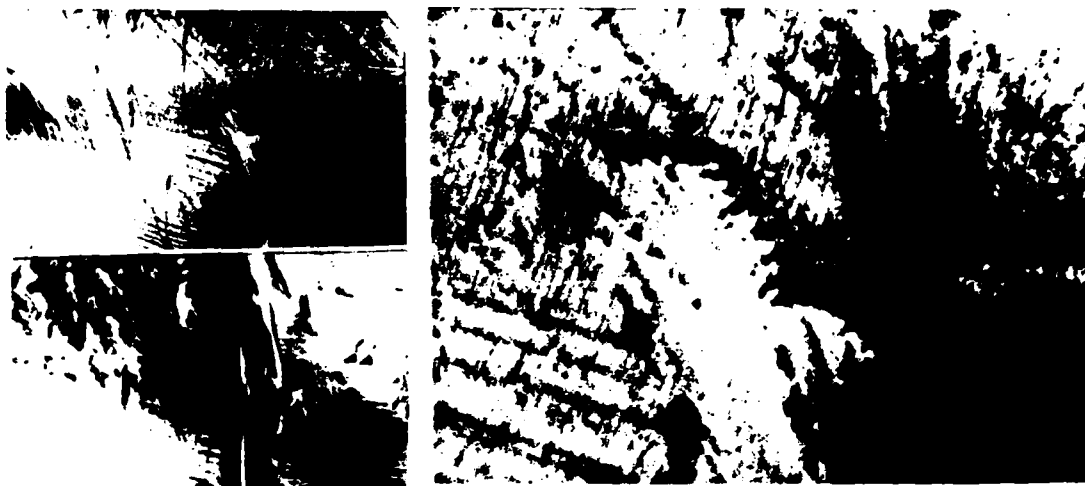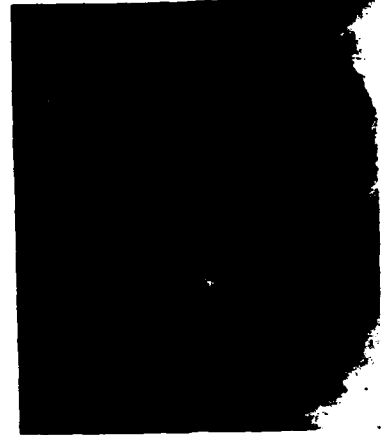
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We have studied plastic deformation in niobium crystals by a variety of x-ray topographic techniques including: Lang topography, double crystal topography and synchrotron white beam topography. The limitations of these direct methods, which rely on the resolution of individual dislocation segments, are illustrated by our results. An indirect topographic method, contour mapping, is described for use with specimens which cannot be profitably studied with the direct techniques. The positions of equi-

Block 20 (cont'd)

inclination contours (analogues to TEM bend contours) are recorded as a function of specimen rotation, allowing measurement of the components of the strain tensor as a function of position on the specimen. Use of monochromatic radiation and of synchrotron white radiation are described and results obtained with contour mapping are summarized. Originator Supplied

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